

COMPARE THE STRUCTURAL AND MAGNETIC SOFTNESS OF NANOCRYSTALLINE ALLOY $\text{Fe}_{74}\text{Cu}_{0.8}\text{Nb}_{2.7}\text{Si}_{15.5}\text{B}_7$ AT ANNEALING TEMPERATURE 580°C AND 650°C FOR DIFFERENT ANNEALING TIME

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ABSTRACT

The nanocrystalline alloys of $\text{Fe}_{74}\text{Cu}_{0.8}\text{Nb}_{2.7}\text{Si}_{15.5}\text{B}_7$ were rapidly solidified and then annealed at temperature 580 (C and 650°C for different annealing time between 1-60 minutes have been studied to observe soft magnetic properties. The grain size, silicon content, lattice parameter of $\alpha\text{-Fe}$ (Si) nanograins are determined for various times with annealing temperature. The highest value of initial permeability has been found within a few minutes at lower annealing temperature. The highest value of permeability of about 43000 has been obtained at lower temperature 580 (C for short annealing time within 1-3 minutes. Permeability does not increase for further annealing in consecutive higher annealing time for the same annealing temperature. At higher annealing temperature, i.e. 650 (C, the value of permeability reduces for short annealing time compare to annealing at 580 (C. For higher annealing temperature and time, there has occurred Si diffusion. Some of the Si diffuses out of the $\alpha\text{-Fe}$ (Si) phase and formed ordered DO_3 phase of Fe_3Si . The magnetic softness of the material deteriorated for the longer annealing time and higher annealing temperature due to the effect of induced anisotropy of the matrix.

KEYWORD: Permeability, Nanocrystalline, Grain Size & Amorphous etc

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INTRODUCTION

A FINEMET alloy with the composition $\text{Fe}_{74}\text{Cu}_{0.8}\text{Nb}_{2.7}\text{Si}_{15.5}\text{B}_7$ exhibiting superior soft magnetic behavior has been derived by Yoshizawa et al. [1]. He has been carried out the extensive research in this composition and then in order to obtain ultra soft magnetic properties, the composition has been varied as well as a heat treatment condition to control the microstructure of the matrix. For the primary crystallization of BCC Fe, the Fe-Cu-Nb-Si-B amorphous alloy has been annealed at temperatures between 500-600°C to achieve a noncrystalline state. In this composition, Cu and Nb combinedly form, ultra fine grain structure to raise excellent soft magnetic properties. The major requirements for soft magnetic properties are high initial permeability and extremely low coercivity which is governed by low or vanishing magneto crystalline anisotropy [2]. The high values of initial permeability of about 10^5 and correspondingly low coercivities of less than 1 A/m provide excellent soft magnetic properties. The nanocrystalline alloy annealed at higher temperature above about 600°C, there is precipitated small fraction of buried compounds like Fe_2B or Fe_3B with the dimensions of 50-100 nm. Magnetic softening should also occur as soon as the structural correlation length which is in the order of the domain wall width.

The aim of the paper is to find out the optimum conditions for the increase of initial permeability as well as soft magnetic properties and compare the nature of soft magnetic behavior when the sample annealed between the lower and higher annealing temperature.

EXPERIMENTAL

Using a single roller melt-spinning technique, amorphous ribbons of nominal composition $\text{Fe}_{74}\text{Cu}_{0.8}\text{Nb}_{2.7}\text{Si}_{15.5}\text{B}_7$ were prepared in air. The ribbons are 6 mm wide and 20-50 μm thick. The ribbons were wound into toroidal cores and they were annealed for different time at temperatures 580 (C and 650 (C. The amorphosity and crystallization products were investigated by X-ray diffraction and X-ray diffraction pattern of the samples was recorded by a Philips X' pert Pro X-ray diffractometer at room temperature with $\text{CuK}\alpha$ radiation.

Using a laboratory built a furnace and Hewlett Packard 4192A impedance analyzer, frequency dependence of complex initial permeability was measured in the frequency range 1 KHz to 13000 KHz.

RESULTS AND DISCUSSIONS

Figure 1 and figure 2 shows the x-ray diffraction pattern of as-cast and annealed samples at 580°C and 650°C with varying annealing time. Since no crystalline phase have formed due to rapid quenching so sharp peaks do not find in the amorphous state. The evolution of crystalline phase appears with the annealing time and have been identified as bcc Fe (Si) using standard software. As the annealing temperature of 580°C, the sharp peaks appear for the annealing time 1 minute shown in Figure 1. As the annealing time increases the peaks become narrower with higher intensity. When the alloys are annealed at a higher temperature of 650°C for the annealing time of 1 minute, it is found that the peak is sharper with a higher intensity than the peak found in the 1 minute which has been measured at the annealing temperature of 580 (C shown in Figure 2. When the annealing time is increased, the peaks become more distinct.

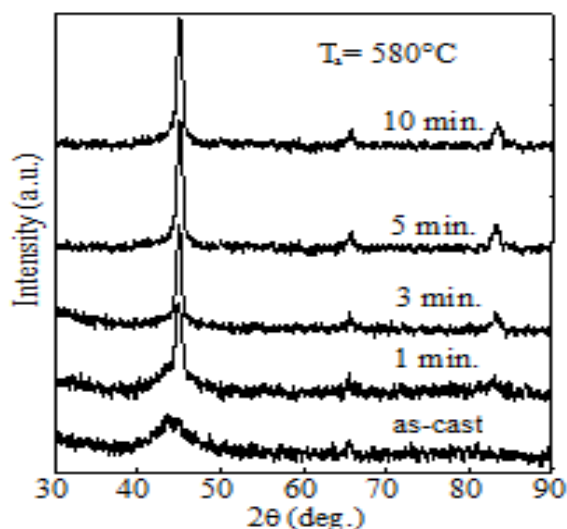


Figure 1: XRD Patterns for different Annealing Time Annealed at 580°C

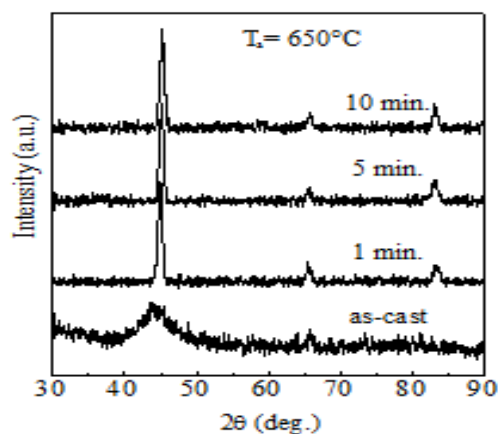


Figure 2: XRD Patterns for Different Annealing Time at 650°C

In Figure 3 and Figure 4 represent the lattice parameters and Si content of α -Fe (Si) nanograins for annealing temperature of 580°C and 650°C. When the sample annealed for successive interval of time, the lattice parameters and Si content of α -Fe (Si) nanograins dispersed in the surrounding amorphous matrix. The lattice parameter has been determined from FWHM of the highest intensity peak using Scherrer's formula and silicon content has been determined from an established quantitative relationship between lattice parameter and silicon content given by Bozorth [3]. From the figure it is shown that the decrease of lattice parameter increases the Si content. The lattice parameter of pure Fe is greater than the lattice parameter of α -Fe (Si), the value of which is 2.866 Å. It is supposed that when the contraction of α -Fe (Si) lattice occurs, then the lattice parameter decreases. It happens because of the diffusion of the silicon with smaller atomic size into the iron lattice of larger atomic size from a substitutional solid solution during the crystallization process to form α -Fe (Si) [4].

When she diffuses into the α -Fe (Si), lattice parameter decreases and again lattice parameter increases due to the sea diffuse out of the α -Fe (Si) lattice [3,5]. It is found that the lattice parameter increases for the subsequent decrease of Si content when the sample annealed above certain time and temperature. It indicates that recrystallization of α -Fe (Si) grains has occurred and silicon diffuses out of the α -Fe(Si) grains during recrystallization.

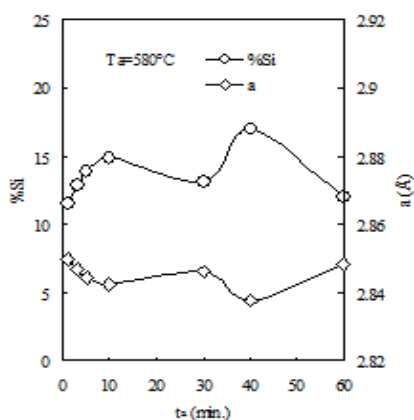


Figure 3: Variation of Lattice Parameter and Si Content at % with Annealing Time at Temperature, $T_a=580^\circ\text{C}$

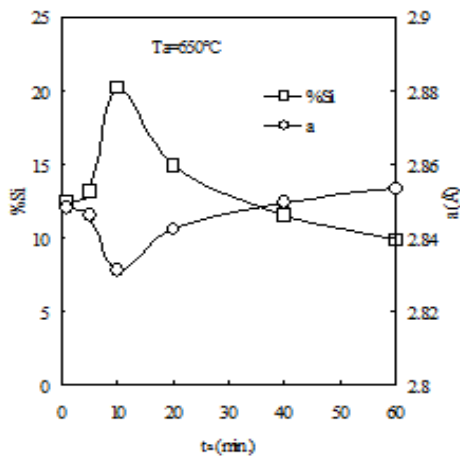


Figure 4: Variation of Lattice Parameter and Si Content at % with Annealing Time at Temperature, $T_a=650^\circ\text{C}$

Figure 5 and 6 represent the mean grain size of the BCC Fe (Si) nanograins using the Scherrer's formula. According to annealing time at $T_a = 580^\circ\text{C}$ is observed as in figure 5 where the limiting value 11.63 nm at 5 minutes remaining constant up to 60 minutes. It is shown from figure 6 that at temperature 650°C , the grain diameter attain the value of 13.45 nm at 1 minute does not change much after subsequent annealing time and for 60 minutes annealing time, the value is 11.95 nm. Effect of annealing temperature and time on grain size can be vividly observed in Figure 5 and figure 6. For the annealing temperature of 580°C , the grain size has changed up to certain annealing time and then almost unaltered. For higher annealing temperature of 650°C , grain size attain for the annealing time of 1 min remain almost constant for subsequent annealing time. This shows that at higher temperature sufficient activation energy of crystallization is attained within a very short time, which is almost at its saturation level and does not change much even after prolonged annealing of 1 HR. This shows that short annealing time and optimum annealing temperature are the main factors for evolution of grain size.

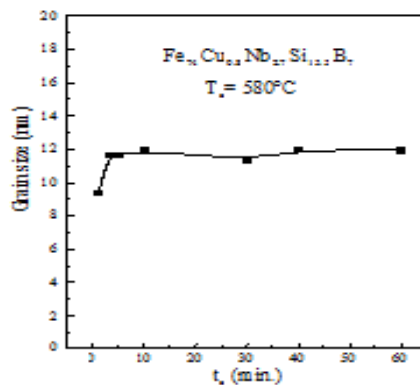


Figure 5: Annealing Time Dependence of Grain Size at 620°C

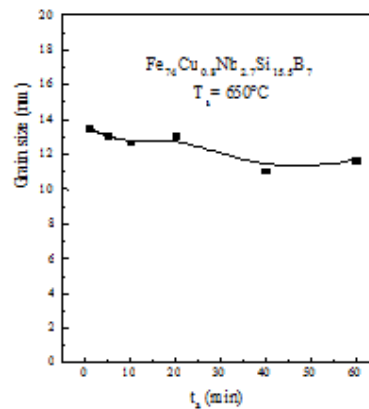


Figure 6: Annealing Time Dependence of Grain Size at 650°C

Figure 7 and 8 show the frequency dependence of the real part of the complex initial permeability μ (up to 10,000 for the as-cast and annealed samples in the temperature 580°C and 650°C for different annealing time. The evolutions of nanocrystalline phase from amorphous precursor are very much time and temperature dependent. Figure 7 represents that at temperature $T_a = 580^\circ\text{C}$, the lowest value of μ (is obtained for 1 minute, which is about 6000 and the highest value of μ (is attained in 3 minutes which is about 43000. Further increase of annealing time, μ (decreases rapidly up to 60 minutes and $\mu' = 11000$ for 60 minutes. At temperature 650°C, it is found that the highest value of μ (is attained at 1 minute and the value is 15000 and then decreases drastically. As the annealing time increases up to 60 minutes, the lowest value is obtained about 1300 as shown in Figure 8. The real part of complex initial permeability decreases due to the formation of Fe-B phase in the amorphous matrix for its higher magneto crystalline anisotropy [6]. The corresponding value of μ'' has been presented in figure 9 and figure 10. To understand the general characteristics of these curves, there is need to explain the permeability spectra of μ' and μ'' . μ' and μ'' are separate into two components at the

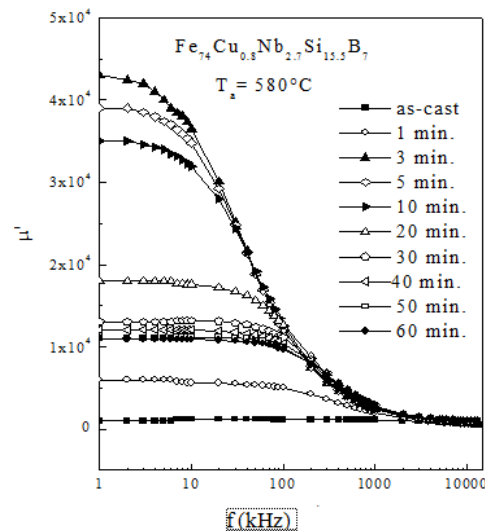


Figure 7: Frequency Dependence of the Real Part of Initial Permeability, μ' for different Annealing Time at Temperature, $T_a = 580^\circ\text{C}$

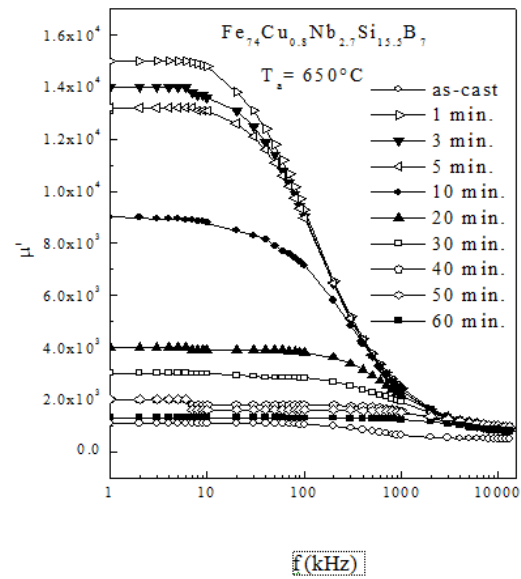


Figure 8: Frequency dependence of the Real Part of Initial Permeability, μ' for Different Annealing Time at Temperature, $T_a=650^\circ\text{C}$

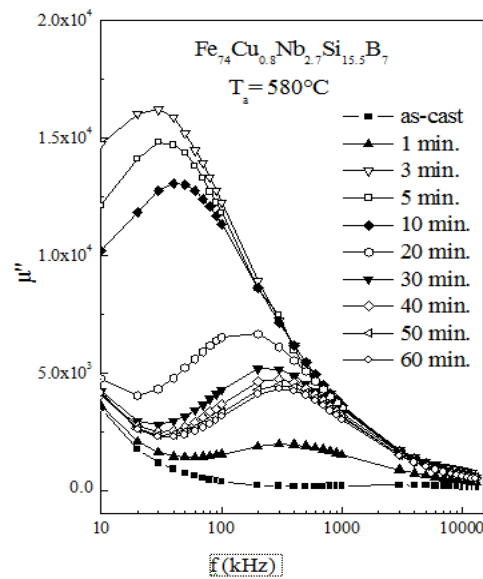


Figure 9: Frequency Dependence of the Imaginary Part of Complex Initial Permeability, μ'' for Different Annealing Time at Temperature, $T_a=580^\circ\text{C}$

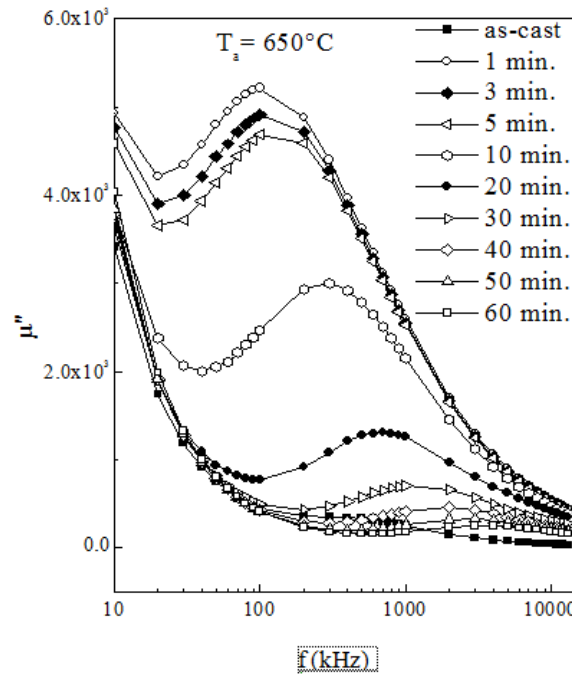


Figure 10: Frequency Dependence of the Imaginary Part of Complex Initial Permeability, μ'' for Different Annealing Time at Temperature, $T_a=650^\circ\text{C}$

High frequency. The permeability with the magnetization represents by μ' and it is called in phase with the alternating magnetic field. Another is μ'' present the imaginary permeability with the magnetization which is known as out of phase with alternating magnetic field [7]. The maxima and minima of the magnetic field, H and that of the induction, B are coincide, we understand from the term “in phase” and by the term “out of phase”, we mean that there is 90° difference between the maxima and minima. The combined complex permeability is given in the complex notation by:

$$\mu = \mu' - j\mu'',$$

Where, μ' = real permeability (in phase), μ'' = imaginary permeability (90° out of phase) and j = unit imaginary vector.

The two permeabilities are after known as permeability dispersion or permeability spectrum. From the above figure, it is seen that the real component of permeability, μ' , is fairly constant with frequency and then falls rapidly at higher frequency. The imaginary component of permeability, μ'' on the other hand increases quite abruptly where the real component, μ' is falling sharply. It appears to reach a maximum at about where the real permeabilities have dropped to about one half of its original value. As the definition of the complex permeability implies, these curves coupled in that the losses is increased due to the frequency increases. As results, the permeability has been found low.

CONCLUSIONS

The main purpose of the experiment was to find out the optimum time and temperature for the formation of nanocrystalline phase and also observe and compare the temperature impact on the magnetic softness at different annealing time. The nanocrystalline alloy of $Fe_{74}Cu_{0.8}Nb_{2.7}Si_{15.5}B_7$ annealed at temperature 580 (C and 650°C for consecutive annealing time up to 60 minutes. The real and complex initial permeability is measured for the temperature 580 (C and

650°C. The best magnetic properties have been observed in the sample annealed at 580°C for 3 minutes and the value is 43000 after that there was decrease the permeability. For annealing time 650 (C, the value of high permeability is 15000 for 1 minute. From this experiment it is found that short time annealing at an optimum annealing temperature above the crystallization temperature provides better soft magnetic properties than for higher annealing temperature and longer annealing time.

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